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I also certify that the attached copy of the request for grant of a Patent (Form 1/77) bears an amendment, effected by this office, following a request by the applicant and agreed to by the Comptroller-General.

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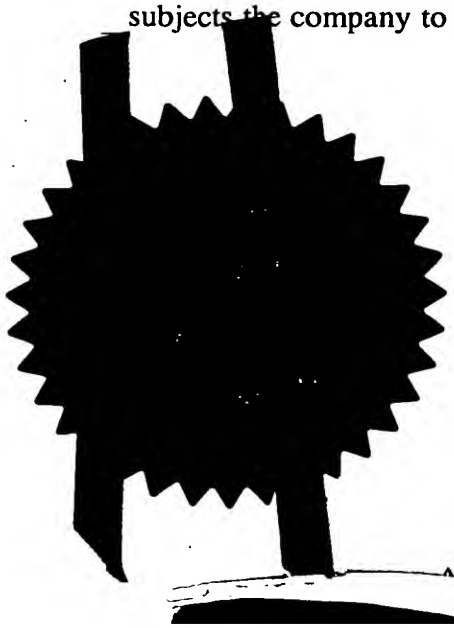

Signed R. Mahoney

Dated 4th August 2000

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1. Your reference

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2. Patent application number
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9917947.5

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

Microtherm International Limited
Hadzor Hall
Droitwich
Worcs WR9 7DJ

Patents ADP number *(if you know it)*

7657711001

If the applicant is a corporate body, give the country/state of its incorporation

United Kingdom

4. Title of the invention

Method of Manufacturing a Thermal Insulation Body

5. Name of your agent *(if you have one)*

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

Derek Jackson Associates
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Request for preliminary examination and search *(Patents Form 9/77)*	1
Request for substantive examination *(Patents Form 10/77)*	
Any other documents *(please specify)*	

11. I/We request the grant of a patent on the basis of this application.

Signature Ian D. Pagan Group Patent Manager Date 29/07/99

12. Name and daytime telephone number of person to contact in the United Kingdom

Ian D Pagan
Tel. 01905 794211

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Method of Manufacturing a Thermal Insulation Body

This invention relates to a method of manufacturing a pressed thermal insulation body. Such a body may be used, for example, as a distance piece or wall in heaters for glass-ceramic cooking appliances, or as insulation in storage radiators.

WO 98/17596 discloses the production of an insulating material particularly for use as walls in heaters for glass-ceramic cooking appliances. The insulating material is based on expanded vermiculite, inorganic binder, microporous material, reinforcing fibres and an infra-red opacifier. The fibres are selected from silica, vitreous silica, R glass, S glass, ECR glass, and similar glasses and mixtures thereof. The reinforcing fibres used contain a maximum of 2 percent by weight of boric oxide and a maximum of 2 percent by weight of alkali metal oxides. Glasses having greater than 2 percent by weight of boric oxide and of alkali metal oxides are excluded as they are said to promote corrosion of heating elements in heaters for cooking appliances.

The insulating material described in WO 98/17596 is said to have better heat insulating properties than are achieved with mica grain particles, such as vermiculite, compressed with a binder and described, for example, in EP-A-204185. However, insulation bodies produced according to WO 98/17596 still have a higher than ideal thermal conductivity, although opacifiers can be added to reduce the thermal conductivity at elevated temperatures.

The addition of a low density microporous material reduces the density of the final moulded body whilst retaining flexural strength and the presence of the reinforcing fibres results in adequate resistance to mechanical damage.

In an alternative approach, as demonstrated in EP-A-560479, a thermal insulation body comprises a low-density microporous material, such as an aerogel, pyrogenic or precipitated silica, and reinforcing glass filaments such as E glass, R glass or S glass. Although such an insulation body has excellent thermal insulation properties, it has less strength than a bonded vermiculite body or a composite body containing vermiculite.

It would be possible to use a higher density material, such as silica fume/volatilised silica, in the production of moulded bodies but these are, at a corresponding density, weaker than bodies made with pyrogenic silica. Furthermore, the thermal conductivity is higher. These materials are to some extent self-opacifying as they contain a small percentage of very well dispersed carbon.

Volatilised silica is produced as a by-product of the manufacture of silicon or ferro-silicon metal. Silica in the form of sand or quartzite is reduced using coal, charcoal, etc. in an electric furnace to form silicon metal. The fumes resulting from this action contain silica and carbon and are collected in hoppers. The chemistry of volatilised silica depends on the type of plant (i.e. silicon or ferro-silicon). The silica content ranges from 84 to 98%, with that of the silica fume produced in silicon metal plants being

between 92 and 98%. The carbon content of the material varies from plant to plant and can range from 0.5 to 6%. Generally the level is around 0.8 to 2%.

The drawback of volatilised silica is its high bulk density when compared to the silica used in a number of microporous insulations. When compressed, the block density has to be correspondingly higher than other forms of microporous insulation. However it is possible to produce handleable blocks at high density which still have acceptable thermal conductivities. Compared to bonded vermiculite or vermiculite composite bodies these mouldings are weak.

We have now found that the strength of thermal insulation bodies based on volatilised silica increases when subjected to heat treatment and the strength shows an unexpectedly rapid increase with increasing temperature.

According to the invention there is provided a method of manufacturing a pressed thermal insulation body comprising the steps of:

1) providing a dry composition comprising:

a) 10 to 100 percent by weight of volatilised silica containing a dispersion of 0.5 to 6 percent by weight of carbon

b) 0 to 40 percent by weight of infra-red opacifier

c) 0 to 50 percent by weight of particulate inorganic filler material

d) 0 to 25 percent by weight of reinforcing filaments

2) pressing the composition to form a body of a desired shape and density; and

3) heating the body to a temperature of between 400°C and 1000°C to effect hardening thereof.

Preferably 80 to 98 percent by weight of the volatilised silica is provided in the composition.

Preferably the carbon content of the volatilised silica is from 0.8 to 2 percent by weight.

During heating of the body the carbon is suitably burnt away such that less than 0.1 percent by weight thereof remains with reference to the volatilised silica.

Preferably the body is heated to a temperature of between 450°C and 800°C.

Preferably 0 to 30 percent by weight of the infra-red opacifier is provided.

The infra-red opacifier may be a material which scatters or absorbs infra-red radiation and may be selected from titanium oxide, iron oxide, mixtures of titanium oxide and iron oxide, zirconium oxide, zirconium silicate, chromium oxide and silicon carbide.

Preferably 0 to 30 percent by weight of the particulate inorganic filler material is provided.

The particulate inorganic filler material may be of low density and may be selected from silica, titania, alumina, vermiculite, perlite, expanded clays and glass microspheres. The silica, titania or alumina may be of a form selected from aerogel, xerogel, pyrogenic and precipitated forms.

Preferably from 2 to 9 percent by weight of the reinforcing filaments are provided.

The reinforcing filaments may be a stable material such as selected from silica, quartz, E glass and modifications thereof, S glass and modifications thereof, R glass, ECR glass, C glass, A glass, ceramic fibre materials, body-fluid-soluble fibres, and mixtures thereof.

The density of the pressed composition may be between 300 and 1200 kg/m^3 and preferably between 500 and 800 kg/m^3.

A number of firing techniques can be used for effecting the necessary heating of the pressed body. Gas or electric powered furnaces or microwave heating equipment are applicable, being arranged to effect a rise in temperature of the body to between 400°C and 1000°C and preferably to between 450° and 800°C. The required heating time depends upon the cross-section of the body.

In addition to an increase of flexural strength of the body with temperature, surface hardness of the body also increases. If only surface hardening is required, a heating time of about 5 minutes at 600°C is all that is necessary for virtually any cross-section of body. However, for full hardening longer heating times may be necessary. The time and temperature of heating is selected by simple experiment whereby the body hardens sufficiently without undergoing any significantly measurable change in dimensions or pore volume.

Although it is not intended that the invention be bound by any specific theory, it is believed that hardening occurs because the volatilised silica particles are coated with carbon soot, in addition to any coarser carbon particles that may be present. In the dry composition state the soot limits the bonding between the silica particles which occurs either by hydrogen bonding or condensation of adjacent Si-OH groups. On heating the material to temperatures at which carbon removal can occur, this soot layer begins to break down. This allows hydrogen bonding and condensation of adjacent Si-OH groups to form bridging oxygens between particles, Si-O-Si. The formation of bridging oxygens would also occur with Si-OH groups from the surface of any glass filaments used as reinforcement, making the reinforcement much more effective. This mechanism could account for the observed increase in flexural strength.

Bodies manufactured according to the invention can be used in a range of applications. For example they may be used as distance pieces or walls in radiant electric heaters, as

bases for storage radiators, as facings for furnaces and incinerators, and as rigid evacuable boards for vacuum use.

For a better understanding, the invention is now described with reference to the following examples.

Example 1 (Comparative)

A composition was produced comprising:

80 percent by weight of micron grade vermiculite supplied by Hoben Davis, UK.

20 percent by weight of water glass K66 binder supplied by Crosfield, UK.

The composition was compressed to provide a dry density of 600 kg/m^3 and dried at a temperature of 300°C for 30 minutes.

Example 2 (Comparative)

A composition was produced comprising:

64 percent by weight of micron grade vermiculite supplied by Hoben Davis, UK.

20 percent by weight of water glass K66 binder supplied by Crosfield, UK.

14.4 percent by weight of pyrogenic silica A200, supplied by Degussa, Hanau, Germany.

1.6 percent by weight of E glass (P201) reinforcing fibres, supplied by Vetrotex. France.

The composition was compressed to provide a dry density of 600 kg/m^3 and dried at a temperature of 300°C for 30 minutes.

Example 3 (Comparative)

A composition was produced comprising:

65.1 percent by weight of pyrogenic silica A200, supplied by Degussa, Germany.

19.6 percent by weight of rutile opacifier, supplied by Tilcon, UK.

5.4 percent by weight of Advantex E glass filaments, supplied by OCF, USA.

9.0 percent by weight of water.

0.9 percent by weight of ammonium bicarbonate, grade FFQ, supplied by Brotherton, UK.

The composition was compressed to provide a dry density of 400 kg/m^3 and dried at a temperature of 150°C for 30 minutes.

Example 4

A composition was produced comprising:

98 percent by weight of fumed silica containing a dispersion of 0.8 to 2 percent by weight of carbon and supplied by VAW, Germany.

2 percent by weight of Advantex E glass filaments, supplied by OCF, USA.

The composition was compressed to form blocks of dimensions 110 mm by 40 mm by 10 mm, for flexural strength testing, and also to form discs of 110 mm diameter and 25 mm thickness, for thermal conductivity testing. The compressed density was 700 kg/m^3. The blocks were fired for 10 minutes at each of a range of temperatures up to 800°C. The discs were fired for 30 minutes at 700°C.

Example 5

A composition was produced comprising:

88 percent by weight of fumed silica containing a dispersion of 0.8 to 2 percent by weight of carbon and supplied by VAW, Germany.

2 percent by weight of Advantex E glass filaments, supplied by OCF, USA.

10 percent by weight of rutile opacifier, supplied by Tilcon, UK.

The composition was compressed to form blocks of dimensions 110 mm by 40 mm by 10 mm, for flexural strength testing, and also to form discs of 110 mm diameter and 25 mm thickness, for thermal conductivity testing. The compressed density was 700 kg/m^3. The blocks were fired for 10 minutes at 700°C and the discs were fired for 30 minutes at 700°C.

The flexural strength, thermal conductivity and Shore A hardness, measured for the samples prepared in Examples 1 to 5, are summarised in the following table.

Example	Flexural strength (kN/m^2)	Thermal conductivity (w/mk) 200°C (mean)	400°C (mean)	Shore A Hardness
1	1550	0.245	0.360	75
2	1450	0.176	0.250	75
3	700	0.029	0.034	55
4 Unfired	480	0.058	0.074	51
4 400°C	480	-	-	67
4 500°C	600	-	-	80
4 600°C	1450	-	-	90
4 700°C	1950	0.081	0.105	98
4 800°C	3200	-	-	98
5 Unfired	420	0.056	0.064	48
5 700°C	1700	0.076	0.089	95

It is seen that in the case of Examples 4 and 5, the method of the invention results in an unexpectedly large increase in flexural strength of the pressed blocks as the firing temperature is increased. With firing temperatures above 600°C, the flexural strength is considerably greater than that of pressed bodies of glass filament reinforced pyrogenic silica (Example 3) and also greater than that of pressed bodies based on vermiculite (Examples 1 and 2).

The thermal conductivity of the pressed discs manufactured according to the method of the invention (Examples 4 and 5) is also seen to be considerably lower than that of the pressed bodies based on vermiculite (Examples 1 and 2).

Furthermore, the pressed bodies manufactured according to the method of the invention and fired, show increasing Shore A hardness values with increasing firing temperature. It is possible to select a short firing time such that only an outer surface skin is

hardened. With increasing firing time, the depth of hardening increases, up to the full depth.

Claims

1. A method of manufacturing a pressed thermal insulation body comprising the steps of:

   1) providing a dry composition comprising:

      a) 10 to 100 percent by weight of volatilised silica containing a dispersion of 0.5 to 6 percent by weight of carbon

      b) 0 to 40 percent by weight of infra-red opacifier

      c) 0 to 50 percent by weight of particulate inorganic filler material

      d) 0 to 25 percent by weight of reinforcing filaments

   2) Pressing the composition to form a body of a desired shape and density; and

   3) heating the body to a temperature of between 400°C and 1000°C to effect hardening thereof.

2. A method according to claim 1, in which 80 to 98 percent by weight of the volatilised silica is provided in the composition.

3. A method according to claim 1 or 2, in which the carbon content of the volatilised silica is from 0.8 to 2 percent by weight.

4. A method according to any preceding claim, in which during heating of the body the carbon is burnt away such that less than 0.1 percent by weight thereof remains with reference to the volatilised silica.

5. A method according to any preceding claim, in which the body is heated to a temperature of between 450°C and 800°C.

6. A method according to any preceding claim, in which 0 to 30 percent by weight of the infra-red opacifier is provided.

7. A method according to any preceding claim, in which the infra-red opacifier is a material which scatters or absorbs infra-red radiation.

8. A method according to claim 7, in which the infra-red opacifier is selected from titanium oxide, iron oxide, mixtures of titanium oxide and iron oxide, zirconium oxide, zirconium silicate, chromium oxide and silicon carbide.

9. A method according to any preceding claim, in which 0 to 30 percent by weight of the particulate inorganic filler material is provided.

10. A method according to any preceding claim, in which the particulate inorganic filler material is of low density.

11. A method according to claim 10, in which the particulate inorganic filler material is selected from silica, titania, alumina, vermiculite, perlite, expanded clays and glass microspheres.

12. A method according to claim 11, in which the silica, titania or alumina is or are of a form selected from aerogel, xerogel, pyrogenic and precipitated forms.

13. A method according to any preceding claim, in which from 2 to 9 percent by weight of the reinforcing filaments are provided.

14. A method according to any preceding claim, in which the reinforcing filaments are selected from silica, quartz, E glass and modifications thereof, S glass and modifications thereof, R glass, ECR glass, C glass, A glass, ceramic fibre materials, body-fluid-soluble fibres, and mixtures thereof.

15. A method according to any preceding claim, in which the density of the pressed composition is between 300 and 1200 kg/m^3.

16. A method according to claim 15, in which the density of the pressed composition is between 500 and 800 kg/m^3.

17. A method of manufacturing a pressed thermal insulation body substantially as hereinbefore described, with reference to Examples 4 and 5.

18. A pressed thermal insulation body whenever manufactured by the method of any preceding claim.

PCT/GB 00/02909

23/77 Aled 28-7-2000

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